00270
Attorney's Docket No.: **KIN61USA**

TRANSMITTAL LETTER TO THE U.S. ELECTED OFFICE
(EO/US) - ENTRY INTO NATIONAL STAGE UNDER 35 USC 371

PCT/JP00/07411	October 24, 2000	November 22, 1999
International Application No.	International Filing Date	Priority Date Claimed

TUNGSTEN TARGET FOR SPUTTERING
Title of Invention

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Applicant(s), Residence Addresses and Citizenship for EO/US

Box PCT
Assistant Commissioner for Patents
Washington, DC 20231
Attn. EO/US

Sir:

Applicants herewith submit to the United States Elected Office (EO/US) the following items under 35 USC 371:

(1) This express request to immediately begin national examination procedures (35 USC 371(f)).

(2) A copy of the international application (in the Japanese Language) including: one cover page, eight pages of specification, one page of claims, two sheets of drawings (FIGs. 1-2), and two pages of International Search Report in the English Language, and two pages of International Search Report in the Japanese Language.

(3) A verified copy of the international application translated into the English Language, including 13 pages of specification, one pages of claims, and two sheets of drawings (FIGs. 1-2).

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JC10 Rec'd PCT/PTO 01 APR 2002

(4) A two page executed Verification of Translation.

(5) An English Language copy of the International Search Report.

(6) An executed Combined Declaration and Power of Attorney.

(7) A copy of the four page Request form.

(8) A copy of the four page Demand for Preliminary Examination.

(9) A copy of the three page Written Opinion dated June 5, 2001.

(10) A copy of the International Preliminary Examination Report dated December 11, 2001.

(11) A First Preliminary Amendment for entry before calculating the filing fee.

(12) A Second Preliminary Amendment, and

(13) Our check in the amount of $890.00 covering the basic national fee as set forth in 37 CFR 1.492(a)(5) (12 claims in total; two independent; and no multiple dependent).

Please charge any additional fees which may be required to effect entry into the National Phase and credit any overpayment to our deposit account 08-3040.

Please direct all communications concerning this application to the undersigned.

Respectfully submitted,
Howson and Howson
Attorneys for the Applicant

By [signature]

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10/089620

JC05 Rec'd PCT/PTO 01 APR 2002

PTO/SB/17 (10-01)
Approved for use through 10/31/2002. OMB 0651-0032
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FEE TRANSMITTAL for FY 2002

Patent fees are subject to annual revision.

TOTAL AMOUNT OF PAYMENT ($) 890.00

Complete if Known	
Application Number	
Filing Date	
First Named Inventor	Fukuyo, et al
Examiner Name	
Group Art Unit	
Attorney Docket No.	KIN61USA

METHOD OF PAYMENT

1. ☑ The Commissioner is hereby authorized to charge indicated fees and credit any overpayments to:
   - Deposit Account Number: 08-3040
   - Deposit Account Name: Howson and Howson
   - ☑ Charge Any Additional Fee Required Under 37 CFR 1.16 and 1.17
   - ☐ Applicant claims small entity status. See 37 CFR 1.27
2. ☑ **Payment Enclosed:** ☑ Check ☐ Credit card ☐ Money Order ☐ Other

FEE CALCULATION

1. BASIC FILING FEE

Large Entity Fee Code	Large Entity Fee ($)	Small Entity Fee Code	Small Entity Fee ($)	Fee Description	Fee Paid
101	740	201	370	Utility filing fee	
106	330	206	165	Design filing fee	
107	510	207	255	Plant filing fee	
108	740	208	370	Reissue filing fee	
114	160	214	80	Provisional filing fee	

SUBTOTAL (1) ($)

2. EXTRA CLAIM FEES

	Extra Claims		Fee from below	Fee Paid
Total Claims		-20** =	X	=
Independent Claims		- 3** =	X	=
Multiple Dependent				=

Large Entity Fee Code	Large Entity Fee ($)	Small Entity Fee Code	Small Entity Fee ($)	Fee Description
103	18	203	9	Claims in excess of 20
102	84	202	42	Independent claims in excess of 3
104	280	204	140	Multiple dependent claim, if not paid
109	84	209	42	** Reissue independent claims over original patent
110	18	210	9	** Reissue claims in excess of 20 and over original patent

SUBTOTAL (2) ($)

**or number previously paid, if greater; For Reissues, see above

FEE CALCULATION (continued)

3. ADDITIONAL FEES

Large Entity Fee Code	Large Entity Fee ($)	Small Entity Fee Code	Small Entity Fee ($)	Fee Description	Fee Paid
105	130	205	65	Surcharge - late filing fee or oath	
127	50	227	25	Surcharge - late provisional filing fee or cover sheet	
139	130	139	130	Non-English specification	
147	2,520	147	2,520	For filing a request for *ex parte* reexamination	
112	920*	112	920*	Requesting publication of SIR prior to Examiner action	
113	1,840*	113	1,840*	Requesting publication of SIR after Examiner action	
115	110	215	55	Extension for reply within first month	
116	400	216	200	Extension for reply within second month	
117	920	217	460	Extension for reply within third month	
118	1,440	218	720	Extension for reply within fourth month	
128	1,960	228	980	Extension for reply within fifth month	
119	320	219	160	Notice of Appeal	
120	320	220	160	Filing a brief in support of an appeal	
121	280	221	140	Request for oral hearing	
138	1,510	138	1,510	Petition to institute a public use proceeding	
140	110	240	55	Petition to revive - unavoidable	
141	1,280	241	640	Petition to revive - unintentional	
142	1,280	242	640	Utility issue fee (or reissue)	
143	460	243	230	Design issue fee	
144	620	244	310	Plant issue fee	
122	130	122	130	Petitions to the Commissioner	
123	50	123	50	Processing fee under 37 CFR 1.17(q)	
126	180	126	180	Submission of Information Disclosure Stmt	
581	40	581	40	Recording each patent assignment per property (times number of properties)	
146	740	246	370	Filing a submission after final rejection (37 CFR § 1.129(a))	
149	740	249	370	For each additional invention to be examined (37 CFR § 1.129(b))	
179	740	279	370	Request for Continued Examination (RCE)	
169	900	169	900	Request for expedited examination of a design application	
Other fee (specify) Basic National Fee as set forth in 37CFR 1.492(a)(5)					890.00

*Reduced by Basic Filing Fee Paid SUBTOTAL (3) ($) 890.00

SUBMITTED BY				Complete (if applicable)	
Name (Print/Type)	William Bak	Registration No. (Attorney/Agent)	37,277	Telephone	215-540-9216
Signature				Date	04/01/2002

WARNING: Information on this form may become public. Credit card information should not be included on this form. Provide credit card information and authorization on PTO-2038.

Burden Hour Statement: This form is estimated to take 0.2 hours to complete. Time will vary depending upon the needs of the individual case. Any comments on the amount of time you are required to complete this form should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, Washington, DC 20231. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Assistant Commissioner for Patents, Washington, DC 20231.

00270
PATENT TRADEMARK OFFICE

KIN61USA

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the Application of:	)	Examiner:
Fukuyo et al.	)	Group Art Unit:
Application No.:	)	
Corresponding International Filing No.: **PCT/JP00/07411**	)	
Filed: Herewith	)	
For: **TITANIUM TARGET FOR SPUTTERING**	)	

Box PCT
Assistant Commissioner for Patents
Washington, DC 20231

FIRST PRELIMINARY AMENDMENT

Sir:

Before calculating the filing fee, please amend the above-identified patent application as follows.

In the Claims:

Cancel multiple dependent claims 3 and 4.

<u>REMARKS</u>

Please enter this preliminary amendment before calculating the filing fee. The multiple dependent claims have been canceled.

Please charge any deficiency or credit any overpayment to our deposit account no. 08-3040.

Respectfully submitted,
Howson and Howson
Attorneys for Applicant

By ______________
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KIN61USA

10/089626

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the Application of:

Fukuyo et al.

Application No.:

Corresponding International Filing No.:
PCT/JP00/07411

Filed: Herewith

For: **TITANIUM TARGET FOR SPUTTERING**

Examiner:

Group Art Unit:

Box PCT
Assistant Commissioner for Patents
Washington, DC 20231

SECOND PRELIMINARY AMENDMENT

Sir:

Please amend the above-identified patent application as follows.

The format of this Amendment complies with 37 CFR §1.121 "Manner of making amendments in applications" as amended on November 7, 2000 pursuant to the "Patent Business Goals Final Rule". Thus, according to 37 CFR §1.121(b)(ii) and (c)(i), amended paragraphs of the specification and amended claims are provided in a form "without markings"; and according to 37 CFR §1.121(b)(iii) and (c)(ii), the amended paragraphs of the specification and the amended claims are also provided, on a separate page, "marked up" to show the changes.

In the Specification:

On page 1, on the line above the title, delete the word "SPECIFICATION".

On page 1, on the first line below the title, replace the subheading "Technical Field" with the subheading --FIELD OF THE INVENTION--.

On page 1, line 16, replace the subheading "Background Art" with the subheading --BACKGROUND OF THE INVENTION--.

On page 4, line 16, replace the subheading "Disclosure of the Invention" with the subheading --OBJECTS OF THE INVENTION--.

On page 4, between lines 21 and 11, insert the subheading --SUMMARY OF THE INVENTION--.

On page 5, line 20, replace the subheading "Best Mode for Carrying Out the Invention" with the subheading --DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT--.

On page 13, line 7, delete the subheading "Industrial Applicability".

In the Abstract:

Please enter the abstract on a separate page as attached.

In the Claims:

Amend claims 1 and 2, as follows. [Format corresponding to 37 CFR §1.121(c)(i), ie. "without markings".]

1(Amended). A titanium target for sputtering comprising a titanium sputtering target in which a concentration of oxygen contained in the titanium target is less than 20ppm and an average grain diameter of said target is 20μm or less.

2(Amended). A titanium target for sputtering comprising a titanium sputtering target in which an impurity concentration of gas components including oxygen, nitrogen and hydrogen contained in the titanium target is 20ppm or less.

Add new claims 5-14, as follows:

5(New). A titanium sputtering target according to claim 1, wherein said target has a Vickers hardness (Vs) of 120 or less.

6(New). A titanium sputtering target according to claim 1, wherein said target has a total content of alkali metals and alkali earth metals including Na and K of 5ppm or less, a total content of heavy metals and light metals of 10ppm or less, and a total content of radioactive elements including U and Th of 1ppb or less.

7(New). A titanium sputtering target according to claim 5, wherein said target has a total content of alkali metals and alkali earth metals including Na and K of 5ppm or less, a

total content of heavy metals and light metals of 10ppm or less, and a total content of radioactive elements including U and Th of 1ppb or less.

8(New). A titanium sputtering target according to claim 2, wherein said target has a Vickers hardness (Vs) of 120 or less.

9(New). A titanium sputtering target according to claim 2, wherein said target has a total content of alkali metals and alkali earth metals including Na and K of 5ppm or less, a total content of heavy metals and light metals of 10ppm or less, and a total content of radioactive elements including U and Th of 1ppb or less.

10(New). A titanium sputtering target according to claim 8, wherein said target has a total content of alkali metals and alkali earth metals including Na and K of 5ppm or less, a total content of heavy metals and light metals of 10ppm or less, and a total content of radioactive elements including U and Th of 1ppb or less.

11(New). A titanium sputtering target according to claim 2, wherein said target has an average grain diameter of 20μm or less.

12(New). A titanium sputtering target according to claim 8, wherein said target has an average grain diameter of 20μm or less.

13(New). A titanium sputtering target according to claim 9, wherein said target has an average grain diameter of 20μm or less.

14(New). A titanium sputtering target according to claim 10, wherein said target has an average grain diameter of 20μm or less.

Version of Amended Claims
with Markings to Show Changes Made
Corresponding to 37 CFR §1.121(c)(ii)

1(Amended). A titanium target for sputtering [wherein] comprising a titanium sputtering target in which [the] a concentration of oxygen contained in the titanium target [for sputtering] is less than 20ppm [or less] and [the] an average grain diameter of said target is 20μm or less.

2(Amended). A titanium target for sputtering [wherein] comprising a titanium sputtering target in which [the] an impurity concentration of gas components [such as] including oxygen, nitrogen and hydrogen contained in the titanium target [for sputtering] is 20ppm or less.

REMARKS

Multiple dependent claims 3 and 4 were canceled in the First Preliminary Amendment. New dependent claims 5-14 are added in this Second Preliminary Amendment. Thus, claims 1, 2 and 5-14 are pending.

The subheadings in the specification have been amended according to standard U.S. practice. No new matter was added.

An abstract has been added on a separate page. The abstract substantially corresponds to the abstract on the cover page of the PCT application. No new matter was added.

Claims 1 and 2 have been amended to better comply with 35 USC §112, second paragraph. No new matter was added.

New claims 5-14 recite the subject matter in canceled multiple dependent claims 3 and 4. No new matter was added.

Applicants respectfully request consideration of claims 1, 2 and 5-14.

Charge any additional fees due to our deposit account no. 08-3040.

Respectfully submitted,
Howson and Howson
Attorneys for Applicant

By _____

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TITANIUM TARGET FOR SPUTTERING

ABSTRACT OF THE INVENTION

A titanium sputtering target that contains a concentration of oxygen in an amount of 20 ppm or less and has a maximum grain diameter of 20 μm or less. The target permits a sputtering operation to be accomplished substantially free from the formation of particles or the occurrence of an abnormal discharge phenomenon. In addition, the target contains a reduced amount of contaminants and is soft.

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the Application of:	
Fukuyo et al.	Examiner:
Application No.:	Group Art Unit:
Corresponding International Filing No.: **PCT/JP00/07411**	
Filed: Herewith	
For: **TITANIUM TARGET FOR SPUTTERING**	

Box PCT
Assistant Commissioner for Patents
Washington, DC 20231

VERIFICATION OF TRANSLATION

Sir:

I, Isamu Ogoshi, having been warned that willful false statements and the like are punishable by fine or imprisonment or both, under section 1001 of Title 18 of the United States Code, and may jeopardize the validity of the above-captioned application and any patent issuing thereon, declare:

(1) I am a patent attorney authorized to practice law in Japan and am engaged in the practice of law with TecLaw Patent & Law Office at Nishi-Shimbashi Sato Bldg. 7F, 4-1, Nishi-Shimbashi 3-chome, Minato-ku, Tokyo 105, Japan.

(2) I am fluent in the Japanese and English Languages.

(3) I have reviewed the attached translation, and certify that it is an accurate English translation of the Japanese language international application of Hideaki Fukuyo,

Yuichiro Shindo and Hideyuki Takahashi, filed on October 24, 2000 and given International Application No. PCT/JP00/07411.

(4) All of the statements made herein of my own knowledge are true and all statements made herein on information and belief are believed to be true.

March 27, 2002
Date

Isamu Ogoshi
Isamu Ogoshi

2/pRTS

SPECIFICATION

TITANIUM TARGET FOR SPUTTERING

Technical Field

The present invention relates to a high-quality titanium target for sputtering capable of significantly reducing the impurity concentration of gas components such as oxygen, nitrogen and hydrogen contained in the titanium target for sputtering, and effectively suppressing the formation of particles upon deposition.

The impurity concentration of gas components such as oxygen, nitrogen and hydrogen described in the present specification will be displayed in mass% in all cases.

Background Art

In recent years, various electronic devices have been devised beginning with the exponential advancement of semiconductors, and the improvement of such functions as well as the development of new devices are being sought on a daily basis.

Under these circumstances, there is an inclination toward the miniaturization of electronic devices and equipment and increase in the degree of integration thereof. Numerous thin

films are formed during the manufacturing process of many of such devices and equipment, and titanium, from its unique metallic property, is used in the formation of thin films of various electronic devices as titanium and its alloy film, titanium silicide film, or titanium nitride film.

Upon forming such titanium (including metal alloys and compounds) films, attention must be given to the fact that the titanium film itself requires a high degree of purity.

The thin films used in semiconductors and the like are made even thinner, shorter and smaller, and, since the mutual distance thereof is extremely small and the integration density is being improved, there is a problem in that the substance forming the thin film or the impurities contained in such thin film will diffuse to the adjacent thin films. This will result in the off-balance of the constituents of the original film and the adjacent films, and there is a significant problem in that the primary function of the film will deteriorate.

In the manufacturing process of this type of thin film, there are cases where the application of heat reaches several hundred degrees, and the temperature also rises during the use of electronic devices equipped with semiconductor devices. This kind of rise in temperature further increases the diffusion speed of the aforementioned substances, and will result in a significant problem where the functions of the electronic devices

will deteriorate.

Moreover, generally speaking, the aforementioned titanium and its alloy film, titanium silicide film or titanium nitride film can be formed with a physical deposition method such as sputtering or vacuum deposition. Explained below is the sputtering method which is used most comprehensively among the above. This sputtering method is a method of physically colliding a positive ion such as Ar^+ to a target disposed on a cathode and releasing the metal atoms constituting the target with the collision energy thereof.

Nitride may be formed by using titanium or its alloy (TiAl alloy, etc.) as the target and conducting sputtering under a mixed gas atmosphere of argon gas and nitrogen.

When impurities exist in the titanium (including alloys and compounds) target upon forming this sputtering film, coarsened particles floating in the sputtering chamber reach the substrate and cause disconnection or short circuit of the thin film circuit, increase the formation amount of particles that cause protrusions on the thin films, and, during sputtering in which gas components such as oxygen, hydrogen and nitrogen exist, cause abnormal electrical discharge thought to be caused by the outburst of such gases. Thus, there is a problem in that a uniform film cannot be formed.

Due to the foregoing problems, it goes without saying that

it is necessary to reduce conventional impurities such as transition metal, high melting point metal, alkali metal, alkali earth metal or other metals. Nevertheless, the aforementioned particles are formed even when decreasing the elements thereof as much as possible, and the current status is that the fundamental solution for such problems is yet to be discovered.

Furthermore, although there are cases where the titanium thin film is used as a pasting layer for preventing the formation of particles upon forming a titanium nitride Ti-N film, the film is hard and sufficient adhesive strength could not be obtained, nor would it function as a pasting layer since it would peel off from the inner wall of the film-forming equipment or its components. Thus, there is a problem in that this would cause the formation of particles.

Disclosure of the Invention

An object of the present invention is to overcome the various foregoing problems, and particularly to provide a titanium target for sputtering free from the formation of particles or occurrence of abnormal discharge phenomenon, having a reduced content of contaminants, and which is soft.

The present invention provides 1) a titanium target for sputtering wherein the oxygen contained in the titanium target for sputtering is 20ppm or less and the average grain diameter

of such target is 20μm or less; 2) a titanium target for sputtering wherein the impurity concentration of gas components such as oxygen, nitrogen and hydrogen contained in the titanium target for sputtering is 20ppm or less; 3) a titanium target for sputtering according to 1) or 2) above, wherein the Vickers hardness (Vs) is 120 or less; and 4) a titanium target for sputtering according to any one of 1) to 3) above, wherein the total content of alkali metal and alkali earth metal such as Na and K is 5ppm or less, the total content of heavy metal and light metal is 10ppm or less, and the total content of radioactive elements such as U and Th is 1ppb or less.

Brief Description of the Drawings

Fig. 1 is a correlation diagram showing the accumulated power and formation status of particles of the Example.

Fig. 2 is a correlation diagram showing the accumulated power and formation status of particles of Comparative Example 1.

Best Mode for Carrying Out the Invention

With the titanium target for sputtering of the present invention, the oxygen contained in the target is 20ppm or less, and the maximum grain diameter of the target is 20μm or less. When the contained oxygen exceeds 20ppm and the maximum grain

diameter of the target exceeds 20μm, as described later, the formation of particles will increase gradually pursuant to the progress of sputtering.

Conventionally, since there is an inclination for the grain diameter to become coarser when reducing the oxygen content, attempts were not made for precisely adjusting the two. Nevertheless, in the present invention, it was discovered that the reduction of oxygen content and the prevention of grain diameter coarsening are particularly effective in preventing the formation of particles.

Moreover, since gas components such as nitrogen and hydrogen other than oxygen existing on the titanium target show similar behavior as oxygen, it is preferable that the total amount of impurity concentration of gas components such as oxygen, nitrogen and hydrogen be 20ppm or less.

Further, when the impurity concentration of gas components such as oxygen, nitrogen and hydrogen is 20ppm or less, the Vickers hardness (Vs) is 120 or less, and a soft film may be formed. By way of reference, the Vickers hardness (Vs) of an ordinary high-purity titanium target is 150, and a considerable fall in hardness can be acknowledged.

Upon manufacturing high-purity titanium, during fused salt electrolysis, it is necessary to sufficiently dry (vacuum drying and dehydration at 300°C or higher) the salt to be used,

and to further store the raw materials and the like under a dry atmosphere.

In addition, the atmosphere during fused salt electrolysis is made into an inert atmosphere such that the outside air will not flow in. If possible, this process should be carried out under an atmosphere wherein the humidity is 30% or less since there is fear of air being mixed in at the time of opening and closing the valve during the extraction of electro-deposited Ti.

Further, during electrolysis, the process is carried out upon making the initial cathode current density 0.6A/cm^2 or less, which is a low current density. It is thereby possible to obtain an electro-deposited Ti having an oxygen amount of 20ppm or less.

The electrolysis temperature is set to 600 to 800°C. If the temperature is less than 600°C, the electro-deposited Ti will become sponge-like, and oxygen will increase. Meanwhile, if the temperature exceeds 800°C, deterioration of crucible materials or the like and evaporation of salt will become more frequent, and productivity will worsen.

Moreover, it is preferable that the electro-deposited Ti obtained from the raw materials is 80% (yield) or less. If the percentage is any higher, this will cause the increase of impurities such as oxygen.

The electro-deposited Ti obtained thereby is cleansed with

purified water (acid cleaning it not preferable as the surface will become oxidized), and, after drying, it is stored in a vacuum or under an inert atmosphere, thereafter pressed under a dry atmosphere, and is further EB (electron beam) dissolved. It is thereby possible to avoid significant increase of oxygen at the time of EB dissolution and to suppress the amount to 20ppm or less.

Next, a billet is prepared by performing hot plastic processing such as hot forging or hot extrusion at 700°C or higher in which dynamic re-crystallization occurs, preferably between 800°C and 950°C, to the EB dissolved ingot having an oxygen concentration of 20ppm or less and to which precise component control has been conducted, in order to break down the unevenness of such ingot and the coarsened cast structure and to make them even and minute.

Cold plastic deformation such as cold forging or cold extrusion is repeatedly implemented to the billet obtained as described above, and, by applying strong deformation to the billet, the crystalline structure of the target ultimately becomes an even and minute structure of 20μm or less.

Then, this billet is cut in order to prepare performs corresponding to the target volume. Cold plastic processing such as cold forging or cold extrusion is further performed to these performs in order to apply strong deformation and to process

the target into a discoid shape or the like.

Moreover, heat treatment at 400 to 500°C is performed for a short period of time to the target having a processing structure accumulated with such strong deformation upon rapidly raising the temperature by using a fluidized bed furnace or the like. A target having a minute re-crystallized structure of 20μm or less is thereby obtained.

Example

Next, an example of the present invention is explained. The present example is merely an example and this invention shall not be limited thereto. In other words, modes or modifications other than the examples included in the scope of technical ideas of the present invention shall be covered in their entirety.

Upon manufacturing high-purity titanium, a chloride bath of sodium chloride (NaCl) -potassium chloride (KCl) was foremost used as the electrolytic bath.

Prior to the use of this electrolytic bath, sufficient vacuum, drying and dehydration were conducted at 500°C for 20hr. Nickel abundant in corrosion resistance was used for the electrolysis vessel and its peripheral equipment in order to avoid mix-in of impurities and to prevent contamination.

Electrolysis was performed at an electrolysis temperature of 740°C, cathode initial current density of 0.3A/cm^2, and under

an Ar atmosphere.

The electro-deposited Ti obtained thereby was cleansed with purified water and, after drying and dehydration, pressed under an atmosphere wherein the humidity is 10% or less.

After the above, electron beam dissolution is performed thereto. Although alkali metal elements will increase due to fused salt electrolysis, this will not be a particular problem since they can be considerably reduced with this electron beam dissolution. Moreover, this will also significantly reduce impurities of gas components such as hydrogen. Since it is necessary to suppress the increase of oxygen as much as possible in this process, this process is performed under a high vacuum.

Radioactive elements U and Th will become condensed in the bath during electrolysis, and will further decrease as impurities of electro-deposited titanium. Next, the EB dissolved ingot was forged and rolled, and rapid heat treatment was performed at 400 to 500°C.

Pursuant to the precise component control in the foregoing process, the concentration of oxygen became 20ppm or less, and the average grain diameter of the material became 20μm or less. After processing this into a target shape, sputtering was performed with an actual production machine, and the accumulated power and the formation of particles were observed.

The results are shown in Fig. 1. Particles on an 8-inch

wafer and of 0.3μm or more were counted.

Although the formation of particles increased slightly from the initial stages of sputtering up to the accumulated power reaching 400kWh, it is clear that the formation of particles has been suppressed and that the state of transition did not change very much. The formation of particles was effectively suppressed by controlling both the oxygen concentration and the grain diameter.

Comparative Example 1

Next, a sputtering target having an oxygen content of 200ppm and in which the maximum grain diameter of the target is 100μm was used, sputtering was performed thereto similarly with an actual production machine, and the accumulated power and formation of particles were observed. The results are shown in Fig. 2.

Although the particles were comparatively suppressed low from the initial stages of sputtering up to the accumulated power reaching 150kWh, sudden burst of particles formation was observed in several places. The formation of particles increased rapidly up to 250kWh thereafter and also became unstable.

Comparative Example 2

A sputtering target having oxygen content of 30ppm and

in which the maximum grain diameter of the target is 15μm was used, sputtering was performed thereto similarly with an actual production machine as in Example 1, and the accumulated power and formation of particles were observed.

The particles were suppressed low from the initial stages of sputtering up to the accumulated power reaching 150kWh, and sudden burst of particles formation was not observed. The formation of particles increased gradually from the vicinity of 300kWh thereafter, and exceeded the standard upper limit of particles, which are 30 particles of 0.3μm or more, at 300kWh.

Comparative Example 3

A sputtering target having oxygen content of 10ppm and in which the maximum grain diameter of the target is 100μm was used, sputtering was performed thereto similarly with an actual production machine as in Example 1, and the accumulated power and formation of particles were observed.

Although the particles were comparatively suppressed low from the initial stages of sputtering up to the accumulated power reaching 250kWh, sudden formation of particles was observed in several places along the way. The formation of particles increased gradually after the sudden formation of particles in the vicinity of 250kWh, and exceeded the standard upper limit of particles, which is 30 particles of 0.3μm or more, at 300kWh.

As described above, a phenomenon of significant formation of particles was observed due to the increase of oxygen and coarsening of grains. In particular, numerous formations of particles were observed when the grain diameter becomes large based on the coarsened titanium grains during sputtering.

Industrial Applicability

Provided is a titanium target for sputtering effective in forming thin films of electronic devices and the like having an advantageous characteristic of being able to considerably reduce the number of particles formed during sputtering by performing sputtering with a high-purity titanium target capable of significantly reducing the impurity concentration of gas components such as oxygen, nitrogen and hydrogen and having a maximum grain diameter of 20μm or less.

CLAIMS

1. A titanium target for sputtering wherein the oxygen contained in the titanium target for sputtering is 20ppm or less and the average grain diameter of said target is 20μm or less.

2. A titanium target for sputtering wherein the impurity concentration of gas components such as oxygen, nitrogen and hydrogen contained in the titanium target for sputtering is 20ppm or less.

3. A titanium target for sputtering according to claim 1 or claim 2, wherein the Vickers hardness (Vs) is 120 or less.

4. A titanium target for sputtering according to any one of claims 1 to 3, wherein the total content of alkali metal and alkali earth metal such as Na and K is 5ppm or less, the total content of heavy metal and light metal is 10ppm or less, and the total content of radioactive elements such as U and Th is 1ppb or less.

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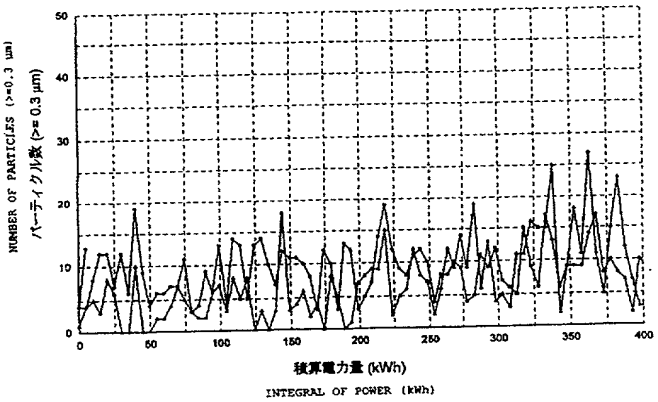
(81) 指定国 (国内): JP, KR, US.

添付公開書類:
— 国際調査報告書

(54) Title: TITANIUM TARGET FOR SPUTTERING

(54) 発明の名称: スパッタリング用チタンターゲット



(57) Abstract: A titanium target for sputtering, characterized in that it contains oxygen in an amount of 20 ppm or less and it has a maximum grain diameter of 20 $(m)m or less. The target allows a sputtering being substantially free from the formation of particles or the occurrence of an abnormal discharge phenomenon, is reduced in the content of contaminants, and is soft.

(57) 要約:

スパッタリング用チタンターゲットに含有する酸素の不純物濃度が２０ｐｐｍ以下、該ターゲットの最大結晶粒径が２０μｍ以下であるスパッタリング用チタンターゲットに関し、パーティクルや異常放電現象が発生せず、汚染物質が少なく、かつ軟質のスパッタリング用チタンターゲットを提供する。

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FIG. 1

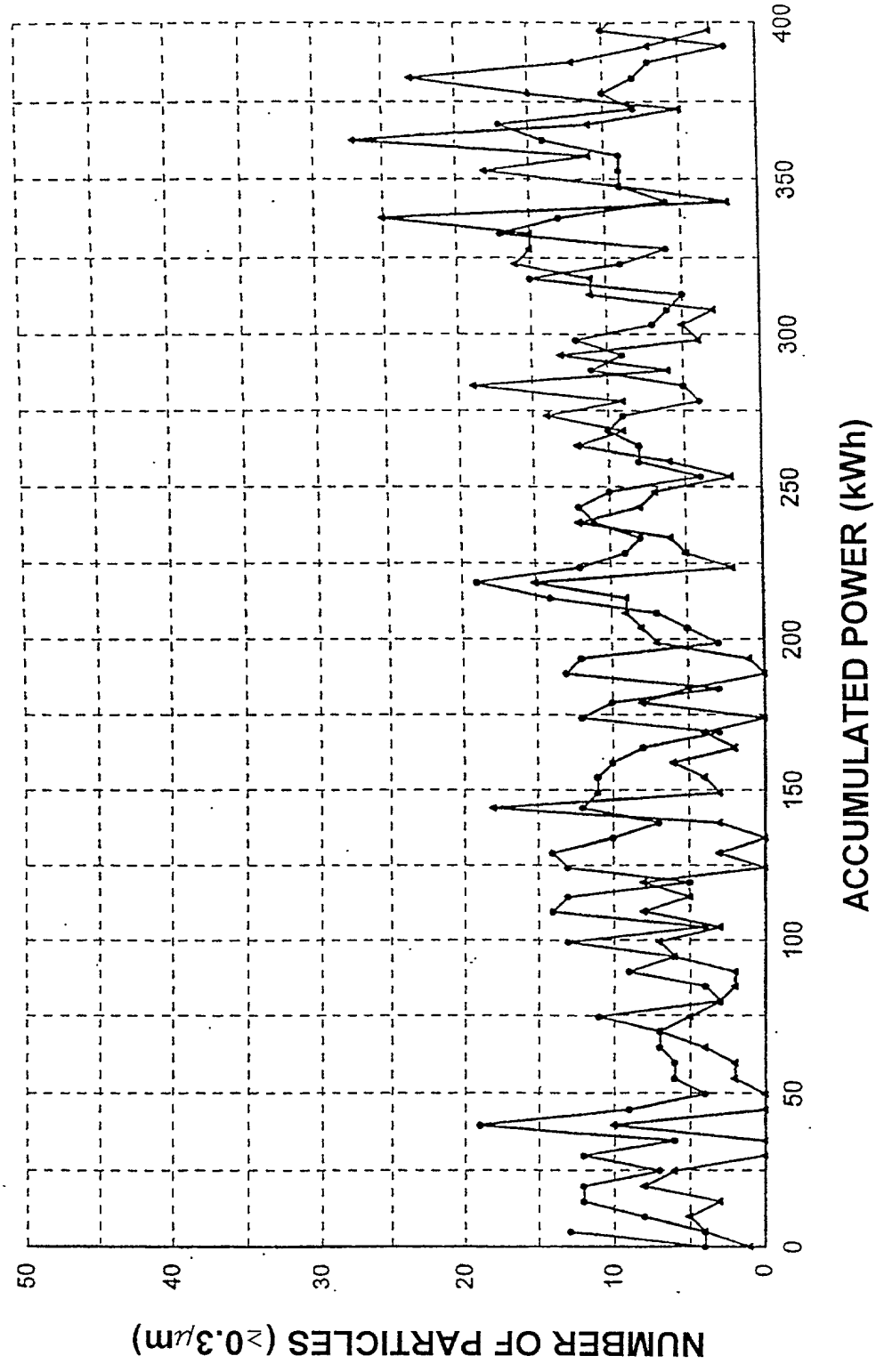
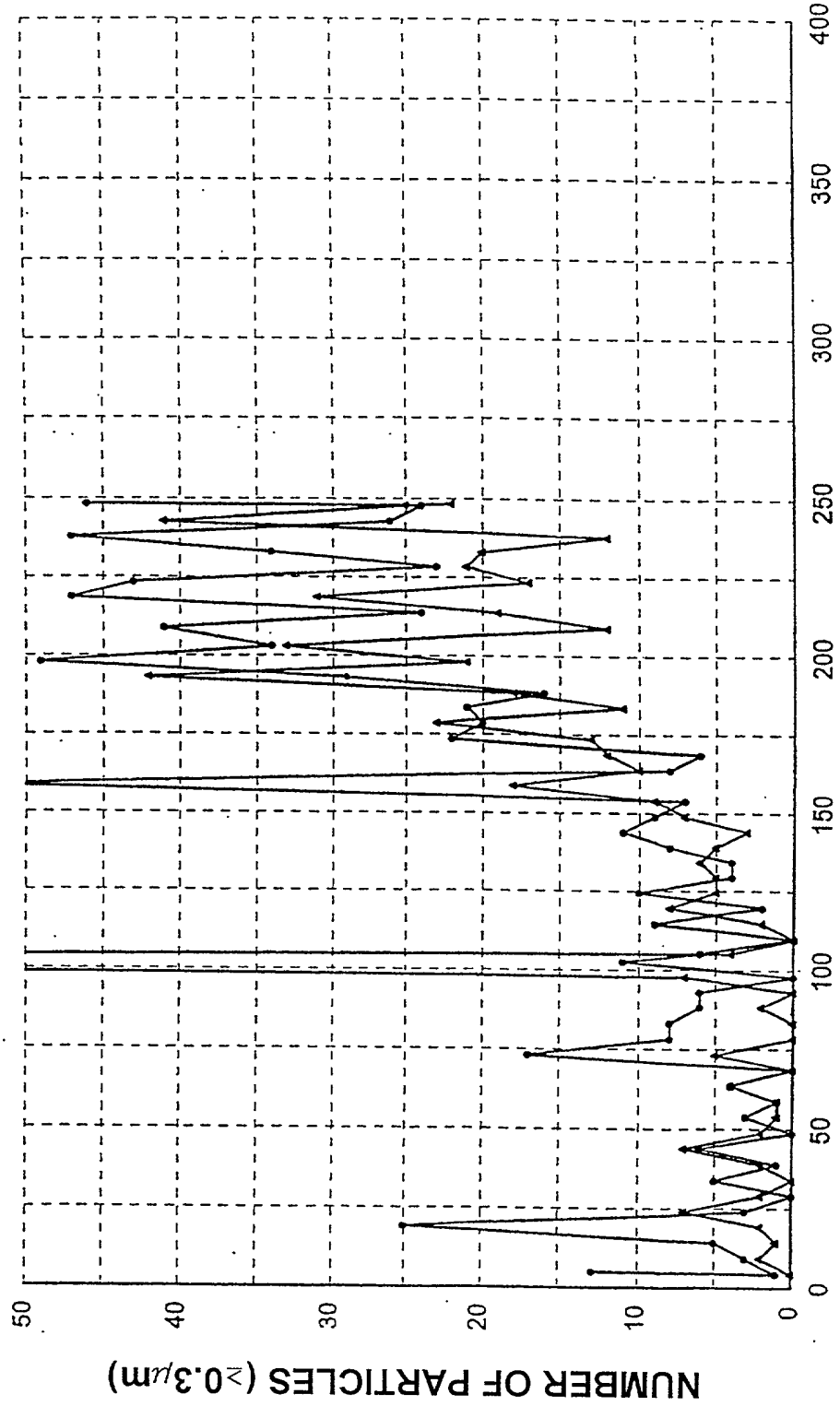


FIG. 2



KIN61USA

DECLARATION AND POWER OF ATTORNEY FOR PATENT APPLICATION

As the below named inventors, we hereby declare that:

Our residences, post office addresses and citizenship are as stated below next to our names, **Hideaki Fukuyo, Yuichiro Shindo** and **Hideyuki Takahashi**. We believe we are the original, first and sole inventors (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled **TITANIUM TARGET FOR SPUTTERING**, the specification of which

(Check one)
____ is attached hereto
____ was filed on _____ as
Application Serial No. _____
and was amended on _____
__X__ was described and claimed in PCT International
Application No. **PCT/JP00/07411** filed on
October 24, 2000.

We hereby state that we have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

We acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, Section 1.56(a).

We hereby claim foreign priority benefits under Title 35, United States Code, Section 119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

Prior Foreign Application(s)

			Priority Claimed	
11-331073	**Japan**	**22 November 1999**	**X**	
(Number)	(Country)	(Day/Month/Year Filed)	Yes	No

We hereby claim the benefit under Title 35, United States Code, Section 120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, Section 112, We acknowledge the duty

to disclose material information as defined in Title 37, Code of Federal Regulations, Section 1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

(Application Serial No.)	(Filing Date)	(Status) (patented, pending, abandoned)

We hereby appoint the following individuals to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith: STANLEY B. KITA, Registration No. 24,561; GEORGE A. SMITH, JR., Registration No. 24,442; MARY E. BAK, Registration No. 31,215; CATHY A. KODROFF, Registration Number 32,980; and WILLIAM BAK, Registration No. 37,277.

Address all telephone calls to William Bak at telephone no. (215) 540-9216. Address all correspondence to HOWSON AND HOWSON, Spring House Corporate Center, P. O. Box 457, Spring House, Pennsylvania 19477.

We hereby declare that all statements made herein of our own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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ADDED PAGE TO COMBINED DECLARATION AND POWER OF ATTORNEY
FOR AUTHORIZATION OF ATTORNEY(S) TO ACCEPT AND FOLLOW
INSTRUCTIONS FROM REPRESENTATIVE

The undersigned to this declaration and power of attorney hereby authorizes the U.S. attorney(s) named herein to accept and follow instructions from:

TecLaw Patent & Law Office
Nishi-Shimbashi Sato Bldg. 7F,
4-1, Nishi-Shimbashi 3-chome,
Minato-ku, Tokyo 105, Japan.

as to any actions to be taken in the Patent and Trademark Office regarding this application without direct communication between the U.S. attorney(s) and the undersigned. In the event of a change in the person(s) from whom instructions may be taken, the U.S. attorney(s) will be so notified by the undersigned.